Novel Magnetic and Chemical Micro Sensors for In-situ, Real-time, and Unattended Use

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ABSTRACT

There exists a need to develop novel, advanced, unattended magnetic and chemical micro-sensor systems for successful detection, localization, classification and tracking of ground time critical targets of interest. Consistent with the underlying long-term objectives of the development of unattended ground sensors (UGS) program we have investigated the use of a new planted ground sensor platform based on Micro-Electro-Mechanical Systems (MEMS) that can offer magnetic, chemical and possibly acoustic detection. The envisioned micro-system will be low-power and low-cost and will be built around a single type of microstructure element integrating a monolithic optical system and electronics package. This micro sensor can also incorporate burst telemetry to transmit the information, a renewable power source and will be capable of operating under field conditions, with sufficient sensitivity to permit high detection rates, and with sufficient chemical selectivity to prevent high false alarm rates. Preliminary studies, initial designs, and key predicted performance parameters will be presented. Possible applications of such as system include sensitive perimeter monitoring such as minefields and military/nuclear bases, vehicle detection, and aircraft navigation systems, and drug enforcement operations. The results of the present work demonstrate that the microcalorimetric spectroscopy technique can be applied to detect and identify chemicals in the ppm level and the studied microcantilever-based magnetometer can provide sensitivities in the order of $1 \mu T$.

Keywords: unattended, micro sensors, low-cost, chemical sensing, magnetic sensing

1. INTRODUCTION

The underlying long-term objective of the UGS program is to support the real-time prosecution of time critical mobile targets, combat vehicles and dismounted soldiers, and to support the characterization of high value man-made structures, surface and underground facilities, with miniature, low power, cost effective, fields of unattended sensors. Of interest are miniature, low power systems that will enhance the aggregate surveillance and tracking capabilities of US forces over potential enemy capabilities in counter detection, counter localization, and counter classification[1-3]. Several state of the art systems have been studied to date with multiple sensor types (acoustic, magnetic, seismic, environmental, and IR) and have demonstrated the ability to detect, track and classify a variety of targets[4]. However, the majority of these systems concentrate on acoustic, seismic and magnetic detection with little or no emphasis on chemical detection. Recently[4] discussed the applicability of UGS on drug enforcement operations. EEG's envisioned microsystem will offer chemical and magnetic detection around a single type of microstructure element.

Although in the first phase of our work we concentrated on hydrocarbon detection and magnetic-field detection, our long term objectives are to expand chemical detection into other areas such as drug and explosives detection as well as expanding the envisioned MEMS system for acoustic, seismic and IR detection. We believe that the integration of these sensing parameters within the same microstructure will significantly contribute to the development of miniature and low cost, yet very capable, UGS.

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The integrated multifunction UGS system will occupy substantially less than a cubic inch. A short description of the magnetic and chemical detection technologies follows.

1.1 Chemical Sensing

Present day chemical detection technologies have been based primarily on adaptation of laboratory instruments. For example, a large number of spectroscopic methods have been introduced over the past twenty years that are based upon methods such as optical adsorption[5-8], laser scattering[9], luminescence[10,11], atomic fluorescence spectroscopy[12] or refractive index changes[13]. However, these methods rely on performing laboratory chemical analyses on extracted samples provide no real-time data for operational feedback and are generally expensive and complicated to use. Currently used miniature, real-time chemical sensors such as multi-analyte, single-fiber, optical sensors[14], sol-gel indicator (SGI) composites[15], portable acoustic-wave sensors (PAWs)[16], thin film resonators[17], and surface acoustic wave array detectors[18] have sufficient chemical sensitivity but suffer from lack of chemical specificity.

In this work we have investigated a new chemical detection technique that can be incorporated into a UGS and affords significant opportunities for higher sensitivity and specificity, miniaturization, mass production and customization based on a microcalorimetric spectroscopy shown schematically in Figure 1. The chemical sensing technique utilizes a wavelength dispersive device and a thermal infrared detector array. During the detection process the sample is allowed to interact/adsorb onto the coated surface of femto-joule sensitive thermal detector. The surface of the detector will be coated with an appropriate chemical layer which preferentially adsorbs/reacts with a category of chemicals similar to the target analytes. As molecules adsorb on the thermal detector surface, various physical changes can take place such as changes in the electrical resistance when a micro-bolometer is used or changes in the bending due to adsorption induced stress when a micro-cantilever thermal detector is used. This step provides the chemical detection sensitivity comparable to recent chemical detection technologies in addition to providing the required selectivity. In order to determine the specific identity of the adsorbed chemical, a photothermal spectrum is obtained by scanning a broadband wavelength region of the detector array with the aid of a wavelength dispersive device. For the wavelengths at which the adsorbed chemical absorbs photons, the temperature of those particular detector pixels will rise proportional to the amount of analyte deposited and heat absorbed. Since different pixels will be exposed to different wavelengths an extremely sensitive and unique photothermal signature response can be determined. The chemical detector surface can be regenerated by ohmic heating of the detector element. This can be achieved with low electrical power since the mass of an element can be very small ($\sim 10^{-9}$ g), and thus its temperature can rise significantly within a very short time, leading to the desorption of analytes from the surface of the thermal detector.
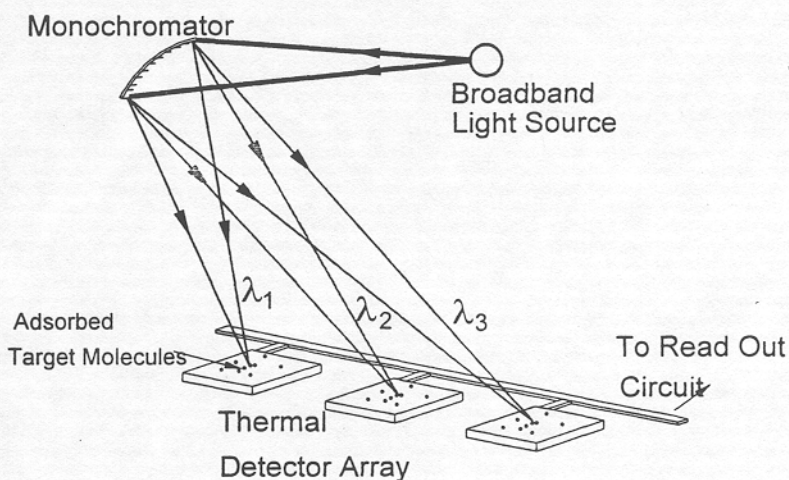

Figure 1. Schematic representation of the principle of microcalorimetric spectroscopy technique. Photons of different wavelength irradiate different thermal detectors configured as a linear array. Molecules adsorbed on the surface of thermal detector elements absorb photons and a photothermal spectrum is obtained.

When target molecules adsorb on the surface of the thermal detector, there will be a dramatic increase in the heat flow to the thermal detector when irradiated with photons that are absorbed by the these molecules. The detection of the thermal signature (photothermal spectrum) provides a sensitivity for our proposed technique in the ppt level and since most chemicals have a distinct photothermal spectrum, they can be easily identified.

1.2 Magnetic Field Sensing Using Micro-Cantilevers

Magnetic sensors are similar to inductive sensors, except a DC magnetic field generated by a permanent magnet is used instead of the AC field. As pickup coils are sensitive only to AC flux, a Hall effect device which is sensitive to DC magnetic fields is often used[19]. The Hall effect is created in a conductive sheet. With a linear current flowing in one axis, a linear field-dependent voltage is measured in the other axis when a magnetic field is induced through the sheet. A drawback of commercially available hall sensors is the large and poorly controlled offset voltage which is compensated by the use of AC coupling or computer calibration strategies[19]. Another type of magnetic sensor with response to DC uses a saturable high permeability material. Permalloy, for example, has a very high permeability, a rectangular hysteresis loop, and low saturation flux density, and can be biased to switch states in response to low level DC magnetic fields. A switching response is also available with a magnet-actuated reed relay, but sensitivity is not as good[19].

Piezoresistive microcantilevers have been used in the past[20,21] as sensitive miniature torque magnetometers to measure magnetic moments as small as 10^{-14} A m^2. [21].

There is increasing interest in the development of miniature magnetometers for detecting and mapping magnetic fields. The trend is constantly toward smaller size, lower power consumption, and lower cost for similar performance. The need for significant improvements in sensitivity, power, and cost is particularly important in applications of unattended ground sensors. Toward this end, recent developments have included the use of cantilevers originally developed for atomic force microscopy (AFM). These type of devices have sensitivities (minimum detectable magnetic field change) that are generally in the range of 1 mT.

Figure 2. Schematic diagram of the bending of a ferromagnetic microcantilever along the z-direction upon exposure to an external magnetic field.

In the present studies the magnetic filed sensing will be achieved by using micro-cantilevers coated with a ferromagnetic layer. However, depositing on a microcantilever layers of materials that exhibit either positive or negative magnetostrictive properties, magnetostriction can be used to detect magnetic fields. Magnetostriction has long been recognized as one of the main energies in the physics of magnetics. It is defined as the change in a length of a ferromagnetic substance - iron, cobalt, nickel, etc.- when it is exposed to a magnetic field. The effect of an external magnetic field B is to produce a differential stress Δs_m

$$\Delta s_m = A_0 E B \tag{1}$$

where A_0 is constant that depends on the specific material and E is the Young's modulus and B is the magnitude of the local magnetic field. Using a bimaterial cantilever (composed of a ferromagnetic cantilever coated on one side with another material such as Si, Au, Al, etc) and applying a magnetic field the ferromagnetic material will respond causing the microcantilever to bend (as shown in Figure 2). By measuring the bending of the cantilever the identification of the magnetic filed will be achieved. When three orthogonal microcantilevers are used to detect magnetic fields both the direction and magnitude of the external field can be determined.
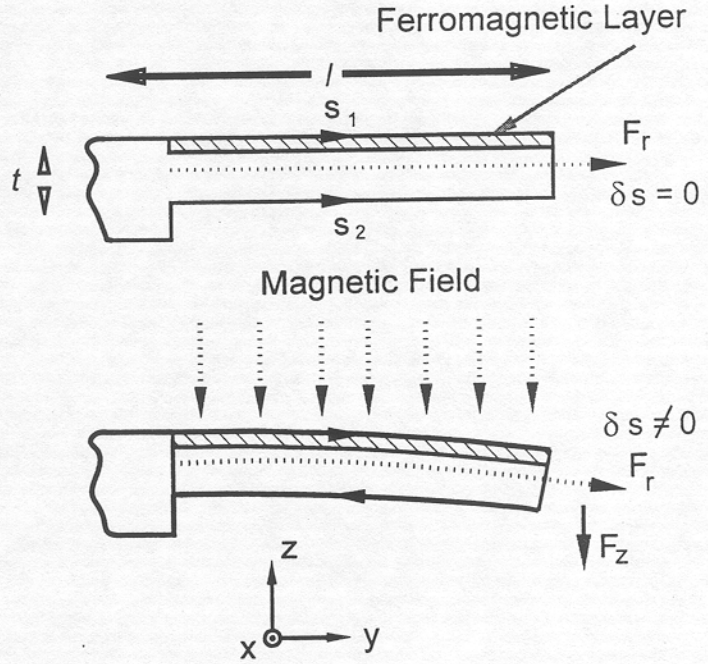
1.3 Microcantilever Bending

The bending of a microcantilever can be extremely sensitive to the sorption of analytes. When the microcantilever is stressed (due to an applied magnetic field) the change in the radius of curvature of the microcantilever is given by[22]

$$\frac{1}{R} = \frac{6(1-\upsilon)}{Et^2}\Delta s \tag{2}$$

where R is the radius of curvature of the microcantilever, υ and E are Poisson's ratio and Young's modulus for the substrate respectively, t is the thickness of the microcantilever and Δs $(= s_1 - s_2)$ is the film's differential stress. The bending of the microcantilever [$z = l^2/(2R)$] is related to the differential surface stress Δs by:

$$z = \frac{3l^2(1-\upsilon)}{Et^2}\Delta s \tag{3}$$

By incorporating equation 1 the microcantilever bending after exposure to magnetic field is expressed as:

$$z = \frac{3l^2(1-\upsilon)}{t^2} A_0 B \tag{4}$$

This results in resonance frequency changes that can be used to detect larger magnetic fields.

2. CHEMICAL DETECTION STUDIES

The chemical detection technique investigated in the present work relies on the adsorption of analytes on the surface of a sensitive thermal detector. The IR detector used in our studies was a microcantilever thermal detector[16-19]. The bending of microcantilevers can be readily determined by a number of means, including optical, capacitive, electron tunneling, and piezoresistive methods. In this work, we used the optical readout technique for observing microcantilever bending due to photothermal heating. The experimental setup used in the present studies is shown schematically in Figure 3. A laser was used in a probe configuration to monitor the microcantilever bending. A HeNe laser (delivering 3 mW at 633 nm) was focused onto the tip of the microcantilever using a 20× microscope objective. In order to minimize heating of the tip by the probe laser, optical power was reduced by placing a neutral density filter with an optical density of 1.0 between the probe laser and the objective. A quad-element photodiode displacement detector was used to collect the reflected probe beam. The bending of the microcantilever depends linearly on the current output ($i_{A,B,C,D}$) of the photodiode. A high narrow bandpass optical filter is placed in front of the photodiode allowing the HeNe laser beam to be detected while preventing other wavelengths from reaching the photodiode. The amplified differential current signal from the quad cell photodiode, $i_{A,B,C,D}$ [$= (i_A+i_B) - (i_C+i_D) / (i_A+i_B + i_C+i_D)$], is monitored and recorded using a digital oscilloscope, or sent to a lock-in amplifier (SR850, Stanford Research Systems) for signal extraction and averaging.

For our initial experiments we used Si triangular microcantilevers that were patterned by creating a serpentine design in each leg utilizing focused ion beam milling. That resulted in a thermal detector of overall effective length of about 500μm and width of 7 μm. Each was 0.6 μm thick and had a 50 nm gold coating uniformly covering one side. Optical read-out as
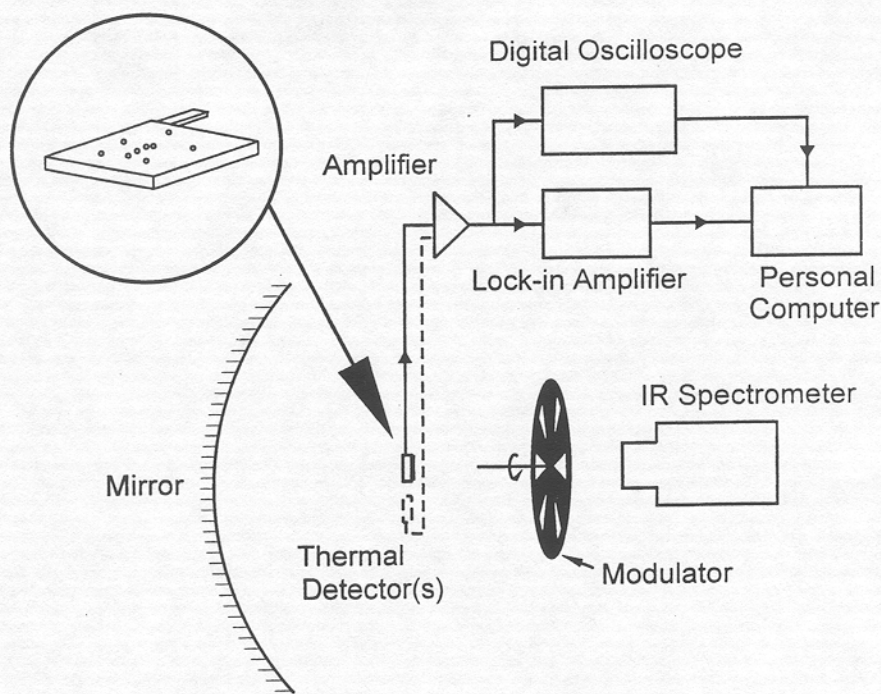
Figure 3. Experimental setup used in the present studies for chemical detection.

described above was used to measure the bending of the microcantilever thermal detector due to photothermal response of molecules adsorbed on its surface. Since the position sensitive photodiode was blind to IR no additional filtering was needed for the read-out circuit. All measurements were conducted at ambient temperature and atmospheric conditions.

The microcantilever thermal detectors were exposed to IR radiation from the spectrometer prior any exposure to chemicals and their thermal response was recorded as a function of photon wavelength. The wavelength region (2.5 to 14.5 μm) attainable with our spectrometer (Foxboro Miran-80 IR spectrometer) was divided into three regions: (i) 2.5 to 4.5 m, (ii) 4.5 to 8.0 μm, and (iii) 8.0 to 14.5 μm. Subsequently, the thermal detectors were exposed to chemical analytes and a new thermal response was recorded as a function of photon wavelength. The increase in thermal response of the detector at particular wavelengths was attributed to the absorption of photons by the molecules adsorbed on the thermal detector surface.

Although our long term objectives are to apply the detection technique discussed in this study to a wide variety of chemicals ranging from toxic organics to explosives, in this phase of our work we concentrated on monaromatic hydrocarbons (i.e., toluene) and PAHs (i.e., anthracene). These toxic organic substances were chosen because of their persistence in the environment, their presence in waste management areas especially military bases, and because of
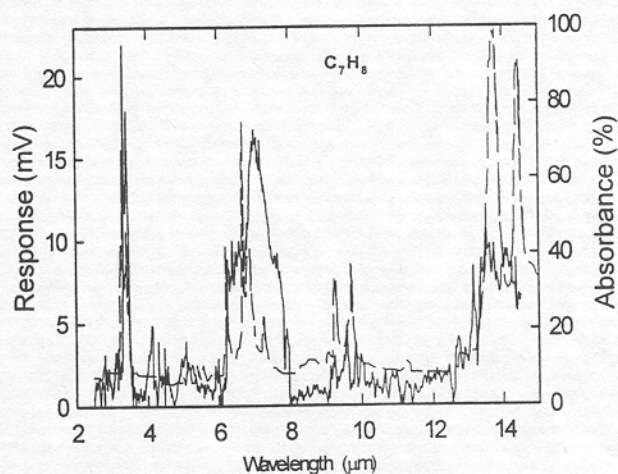

Figure 4. Photothermal response of a microcantilever thermal detector exposed to toluene as a function of photon wavelength (solid curve). The dashed curve represents the infrared absorption spectrum of toluene[23].

their high volume of annual release in the air.

In Figure 4 we plotted the thermal response of a microcantilever thermal detector from 2.5 µm to 14.5µm with toluene molecules adsorbed on its surface (solid curve). The photothermal spectrum was obtained with about one monolayer of coverage. Also plotted in Figure 4 is the IR adsorption spectrum[23] of toluene between 2.5 and 14.5µm (dashed curve). It can be seen that the photothermal spectrum exhibits peaks that correspond well with the IR adsorption peaks. In Figure 5 the photothermal response of a thermal detector exposed to anthracene and the related IR spectrum found in the literature are presented.
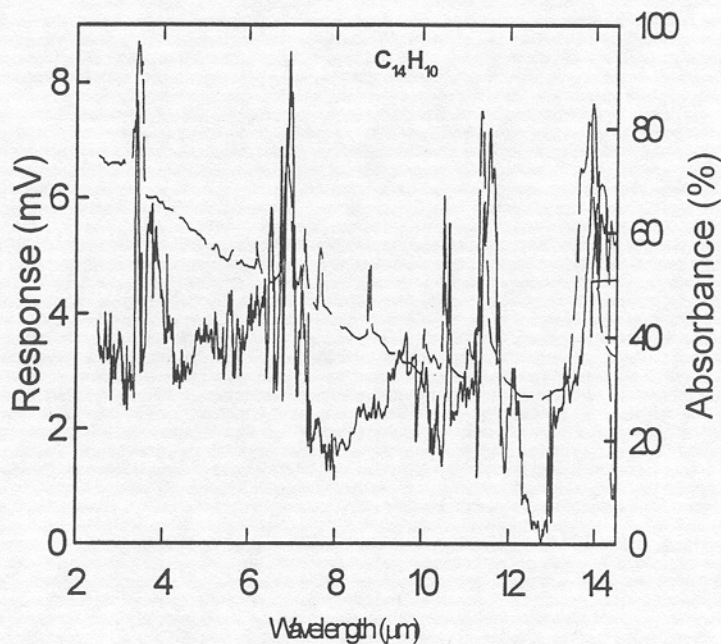

Figure 5. Photothermal response of a microcantilever thermal detector exposed to anthracene as a function of photon wavelength (solid curve). The dashed curve represents the infrared absorption spectrum of anthracene[23].

3. MAGNETIC FIELD DETECTION STUDIES

In this work we describe simple, small, light weight, low cost, and low power consumption ferromagnetic microcantilever-based magnetometer to **measure vector magnetic fields**. We believe that the chosen device has a wide dynamic range such that it can measure magnetic fields over both the nanotesla and tesla regimes. Figure 4 shows a schematic diagram of the cantilever magnetometer. In the measurements described below, the device is placed perpendicular to a magnetic filed generated by an electromagnet. The applied magnetic field causes the cantilever to deflect from its equilibrium position proportionally to the vector component of the field perpendicular in the plane of the cantilever. This amplitude is measured using the deflection of a dc driven diode laser beam reflected from one of the free ends of the bar onto a position sensitive detector (PSD). The difference in outputs was fed into a lock-in amplifier whose reference signal is obtained from the cantilever drive wave form. The range of sensitivities capable of being measured by such a magnetometer can be readily adjusted by suitable alteration of the drive current. A commercial magnetometer (Gaussmeter Model 5070, from F.N. Bell) served as a calibration source. The magnetometer has a quoted field measurement range of 0.01 mT to 1.99 T. The microcantilever used was silicon

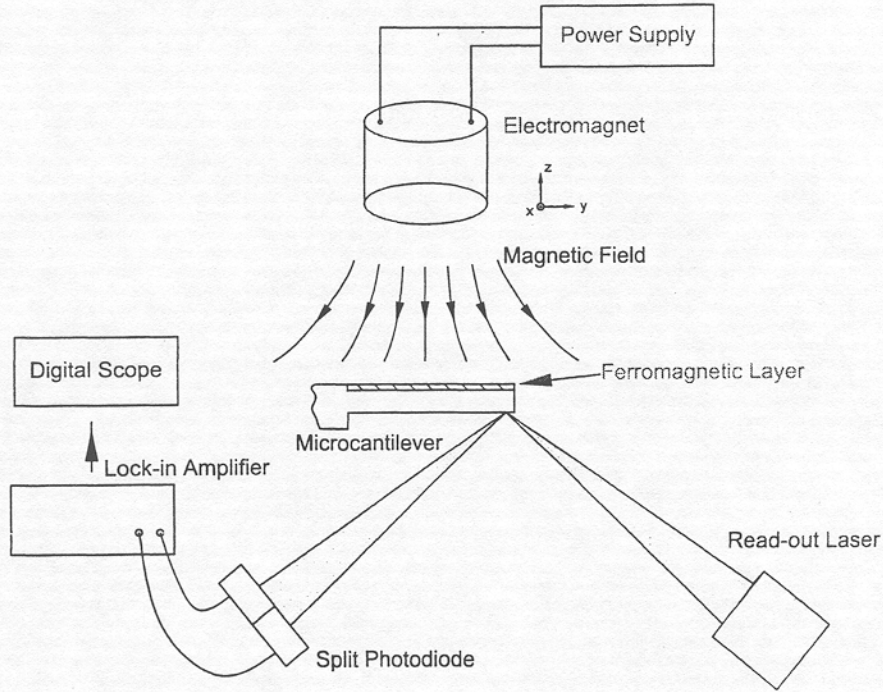gold coated with a length of 180μm, a width of 38μm and a bending force constant $k = 0.40$N/m.

Figure 6. Experimental setup used in the present studies for magnetic field detection.

In Figure 7 we plotted the time evolution of the microcantilever deflection when exposed to a magnetic field. The time t=0 corresponds to the time measurements started. It can be seen that after the introduction of the magnetic field, a rapid response of the microcantilever bending is observed. We should point out that with no further improvements a minimum sensitivity of ~ 2.5 μT was achieved. We believe that careful optimization of the microstructure will lead to a sensitivity at least 1 nT. The responses are given in arbitrary units since they represent the output of the lock-in amplifier. The absolute response is dependent on several variables including laser power and geometry of the optical system. In the setup used in these experiments the output of the PSD was typically on the order of 0.4 mV/nT rms. It is apparent from these results that the response of the cantilever magnetometer device is linear over these ranges and that its sensitivity is at least 1 nT.
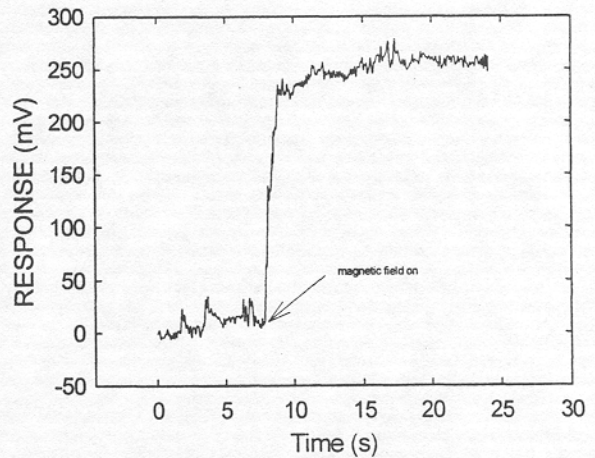

Figure 7. Microcantilever response due to exposure to a magnetic field.

4. CONCLUSIONS

The results of our present studies demonstrate that a microcantilever based system represents an important development and could be used in an UGS to detect magnetic fields and the presence of target chemicals. We have illustrated that the microcalorimetric spectroscopy technique can be applied to detect and identify chemicals in the ppm level and the studied microcantilever-based magnetometer can provide sensitivities in the order of $1\mu T$. Microcantilevers represent an important development in UGS and can be expected to provide the basis for considerable further developments. Low-cost, high-performance, modular, miniature unattended sensors will expand the commercial markets for home and industrial security systems, industrial process monitoring systems, and environmental monitoring systems. The microcantilever based chemical and magnetic microsensor presented in this studies will incorporate high performance optics, telemetry, and power source and will be capable of operating under field conditions, with sufficient sensitivity to permit high detection rates, and with sufficient selectivity to prevent high false alarm rates.   Our future work will involve optimization of the cantilevers used for magnetic field detection, studies on several parameters that could influence the magnetometer's performance, and integration of the chemical and magnetic detection systems.

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